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Designation: F 950 – 02

Standard Test Method for Measuring the Depth of Crystal Damage of a Mechanically Worked Silicon Slice Surface by Angle Polishing and Defect Etching[1]

This standard is issued under the fixed designation F 950; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method describes a technique to measure the depth of damage, on or beneath the surface of silicon wafers prior to any heat treatment of the wafer. Such damage results from mechanical surface treatments such as sawing, lapping, grinding, sandblasting, and shot peening.

1.2 The principal application of this test method is for determining the depth of damage of the non-polished back surface that has had intentionally added work damage.

1.3 The measurement is destructive since a specimen is prepared from a section of a silicon wafer.

1.4 Depth of damage can be measured in the range of 5.0 to 200 µm using this method.

1.5 This test method is intended for use in process control where each individual location is resposible to determine the internal repeatability to its satisfaction.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific warnings and hazard statements are given in 8.6 and Section 9.

2. Referenced Documents

2.1 *ASTM Standards:*

D 5127 Guide for Ultra Pure Water Used in the Electronics and Semiconductor Industry[2]

E 122 Practice for Choice of Sample Size to Estimate a Measure of Quality for a Lot or Process[3]

F 532 Test Methods for Measuring Width of Defects in Optical Surfaces, Using Nomarski Differential Microscopy[4]

F 672 Test Method for Measuring Resistivity Profiles Perpendicular to the Surface of a Silicon Wafer Using a Spreading Resistance Probe[5]

2.2 *SEMI Standard:*

C28 Specifications and Guidelines for Hydrofluoric Acid[6]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *damage*—a defect of the crystal lattice of a single crystal silicon specimen in the form of irreversible deformation. The damage is the result of mechanical surface treatments such as sawing, lapping, grinding, sandblasting, and shot peening at room temperature without subsequent heat treatments.

3.1.2 *damage-free polishing*—a method of preparing a surface of a silicon specimen without creating any mechanical damage detectable by this method.

3.1.3 *bevel angle* (α)—the smaller of the angles between the wafer surface and the section plane. (See Fig. 1.)

3.1.4 *damage depth* (T_z)—the maximum thickness of the damage region. The damage is revealed by a preferential etch that removes silicon in the region of the deformation. Preferential etching occurs because the chemical potential in the region of the deformation is changed by the stress fields associated with the deformation. The depth of damage is expressed in micrometers.

4. Summary of Test Method

4.1 A silicon specimen is coated with silicon nitride by a low-pressure plasma method to a minimum thickness of 1 µm. The specimen is then beveled at a small angle by a polishing technique that produces no additional mechanical damage. The bevel angle is measured. The beveled specimen is etched to reveal the damage. The length of the damage region is measured from the beveled edge on the beveled section. The

[1] This test method is under the jurisdiction of ASTM Committee F01 on Electronics and is the direct responsibility of Subcommittee F01.06 on Silicon Materials and Process Control.

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[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

[4] Discontinued; see 1993 *Annual Book of ASTM Standards*, Vol 06.01.

[5] *Annual Book of ASTM Standards*, Vol 10.05.

[6] Available from the Semiconductor Equipment and Materials International, 3081 Zanker Road, San Jose, CA 95134 (www.semi.org).

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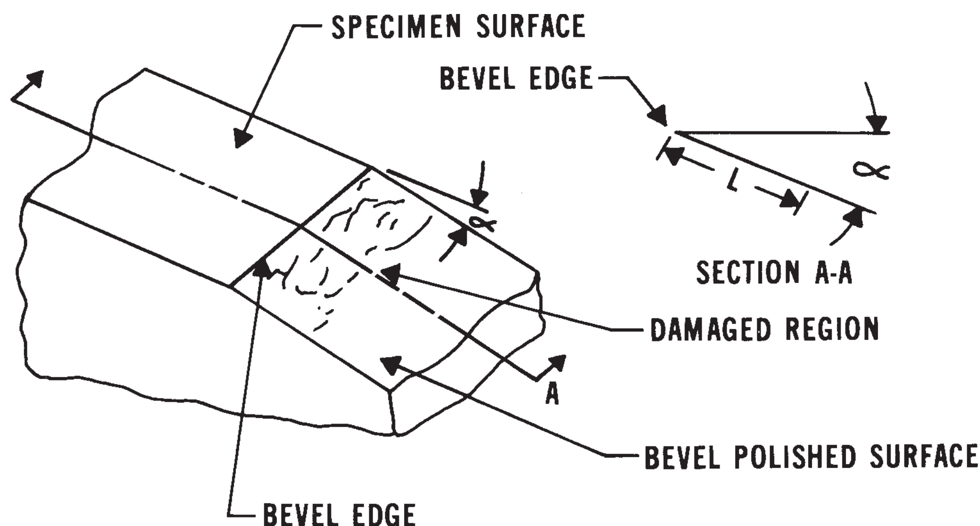

NOTE 1—A 1-μm thick LPCVD nitride film is deposited on the specimen surface prior to beveling.

FIG. 1 Bevel Polished Specimen

depth of damage is then calculated from the relationship between the measured damage length and the sine of the bevel angle.

5. Significance and Use

5.1 This test method provides a means for measuring the depth of mechanical damage in silicon wafers in the range from 5 to 200 μm.

5.2 This test method can be used for process control or research and development purposes. It is not recommended for use in material acceptance.

6. Interferences

6.1 *Choice of Bevel Angle*—A bevel angle must be used such that a magnification of the depth of damage is at least a factor of 5, or the damage may not be detected. Bevel angles

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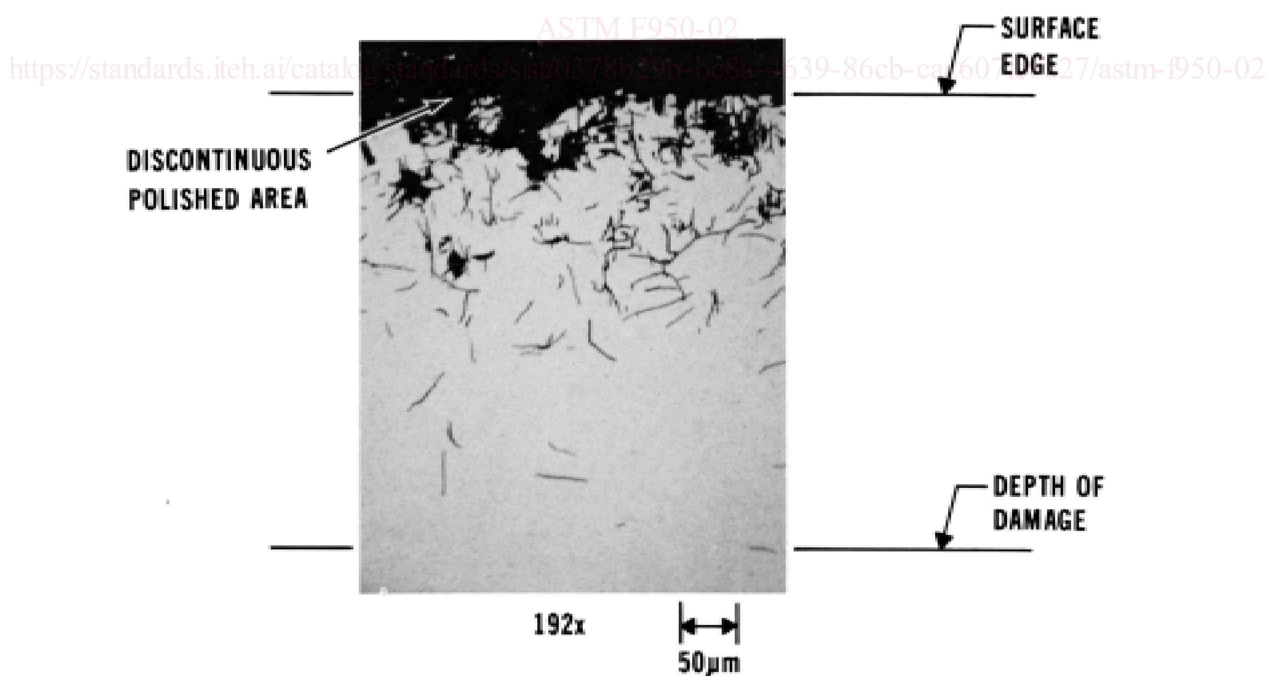

NOTE 1—The surface damaged from sandblasting.

NOTE 2—The 1-μm thick LPCVD nitride film is not visible in the photomicrograph.

FIG. 2 5°44′ Bevel Angle Polished Surface After 1-min Defect Etch

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less than 5°44 min are not recommended because of difficulty in determining the surface edge due to the uneven surface topography generated by the damage. (See Fig. 2.) Table 1 lists the relationship of bevel angle (α), bevel length (*L*), and damage depth (T_z).

6.1.1 Even with a 5°44 min angle, there may be difficulty in determining the bevel edge for surface damage that generates a very rough surface. The bevel edge can be determined by the apparent "discontinuous" polished areas. (See Fig. 2.)

6.2 Damage depth may be nonuniform over a whole wafer area. Because the sample specimens are relatively small with respect to the whole wafer area, depth of damage variations may not be detected; thus, the measurement of the damage depth may be ambiguous.

6.3 Measurement of the bevel angle must be done after lapping and *before* polishing, otherwise the correct angle may not be measured. The alkaline polishing compound may cause a slight surface perturbation near the silicon–nitride interface and at the edges of the specimen.

7. Apparatus

7.1 *Apparatus to Bevel Polish the Test Specimen*:

7.1.1 *Beveling Jig*, consisting of a solid cylinder that is free to move within a hollow cylinder. The specimen is wax mounted onto a beveled sample mount which is then attached to the free moving cylinder as shown in Fig. 3.

7.1.2 *Hot Plate, Diamond Scriber, and Tweezers*.

7.1.3 *Polishing Equipment* that can vary the polishing pressure and will not produce crystal damage.

7.2 *Cement Removal*—The usual chemical laboratory apparatus such as beakers. Adequate facilities for handling and disposing of chlorinated solvents and their vapors are essential.

7.3 *Optical Measurements*:

7.3.1 *Reflection-Light Microscope* with mechanical stage and Nomarski interference contrast optics capable of 100 to 500× magnification as specified in Test Methods F 532.

7.3.2 *Stage Micrometer*.

7.4 *Apparatus to Measure the Angle Beveled on Silicon Specimen*—See Appendix of Test Method F 672.

7.5 *Hydrofluoric Acid-Proof Chemical Laboratory Apparatus*, such as fluorocarbon, polyethylene, or polypropylene beakers, graduates, pipets, and tweezers.

7.6 *Acid Sink, in a fume hood*, with facilities for disposing of acids and their vapors.

7.7 *Facility for Low-Pressure Plasma Nitride Deposition*—(Plasma enhanced CVD SiN capable of 1 µm thick film at ~330°C deposition temperature.)

TABLE 1 Relation of Bevel Angle (α), Bevel Length (*L*), and Damage Depth (T_z)

Angle (α)	Sine (α)	T_z, µm	5	10	100	200
			Bevel Length (*L*), mm			
17 min 11 s	0.005		1	2	20	40
34 min 23 s	0.01		0.5	1	10	20
1°9 min	0.02		0.25	0.5	5	10
2°52 min	0.05		0.01	0.2	2	4
5°44 min	0.10		0.05	0.1	1	2
11°32 min	0.20		0.025	0.05	0.5	1

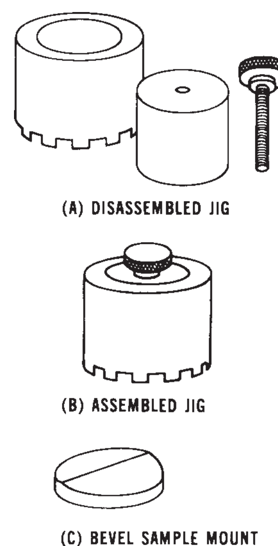

FIG. 3 Lapping/Polishing Jig and Sample Mount

8. Reagents and Materials

8.1 *Purity of Reagents*—All chemicals shall conform to the assay and impurity levels of Grade 1 SEMI Specifications where they exist. Reagents for which SEMI specifications have not been developed shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society,[7] where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 *Purity of Water*—Reference to water shall be understood to mean Type E-3 or better water as described in Guide D 5127.

8.3 *Lapping Compounds*—0.1 µm diamond slurry or 3.0 µm silicon carbide wet/dry lapping paper.

8.4 *Polishing Compound*—Alkaline suspension of colloidal amorphous silica of sub-micron particle size with pH of 10 to 12.

8.5 *Polishing Pad*—Polyurethane based poromerics. These pads are commercially available from various metallurgical polishing supply companies.

8.6 *Mounting Cement*—Glycolphthalate or equivalent mounting cement with similar melting point and solubility in trichlorethane or perchlorethane. **Warning**—These chlorinated solvents are on the suspected carcinogen lists of NIOSH.

8.7 *Mounting Cement Solvent*—Perchlorethane or trichlorethane.

8.8 *Wipers*, residue-free, non-scratching.

8.9 *Brush* with soft nylon bristles.

8.10 *Detergent*, non-ionic 0.25% solution by volume.

8.11 The chemicals used for defect delineation shall have the following nominal assay: Chromium Trioxide >98%; Hydrofluoric Acid, concentrated 48.8 to 49.2%.

[7] "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Analar Standards for Laboratory U.K., Chemicals," BDH Ltd., Poole, Dorset, and the "United States Pharmacopeia."

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